

Advances in New Durable Heat-Resistant Cement System and Its Application in Hot Dry Rock

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ABSTRACT

It is easy to cause the strength retrogression of cement sheath, especially at the long-term high temperature condition of dry hot rock. At present, the method of adding sand to strengthen cement is widely used in high temperature cementing operations. However, the strength retrogression of cement still exists when the temperature is above 160°C, resulting in casing collapse and annular flow, which seriously affects the wellbore safety. To solve the problem of cement strength retrogression at the ultra-high temperature, a new high-temperature stabilizer (SCKL) was developed by optimizing the phase composition of cement based on the enhanced effect of silica aluminum bonding. The effect of SCKL on the inhibition of set cement strength retrogression at high temperatures was evaluated. The influence of temperature on the microstructure and hydrates of set cement was revealed based on the SEM and XRD tests. The results showed that the compressive strength of set cement was 18.2 MPa at 48-hour age for 300°C curing temperature. And the strength reached 23.2 MPa after 30 days, which inhibited the strength retrogression of cement under long-term high temperature conditions. Finally, a new durable heat-resistant cement system was developed. The cement slurry had very good settlement stability and could meet the requirement of safe cementing in DHR wells at a temperature difference of 100°C. The durable heat-resistant cement system has been successfully applied in GH-01 to GH-05 Wells in Gonghe Basin, Qinghai Province. The cementing qualities are very good, which provides a guarantee for the integrity of cement sheath sealing in dry hot rock.

1. INTRODUCTION

The dry hot rock (DHR) resources in China are commonly buried between 3500 m and 7500 m. The rock layer temperature may be as high as 150 to 250°C, containing about 6.3×10^6 EJ of heat energy, and the recoverable heat energy is about 1.26×10^5 EJ, equivalent to 1320 times of China's total energy consumption in 2010. So there's a great prospect for the development of these resources [1]. At Qinghai Basin in China, the cementation job in DHR wells is faced with some troubles like high bottomhole static temperature (>150°C), large cemented interval temperature difference (>60°C), fluctuation in wellbore temperature and pressure difference during production, etc. [2-3], which have led to cementing difficulties like strength retrogression at the bottom of set cement.

At present, the main method to inhibit strength retrogression of set cement at high temperatures is to increase the content of silica powder (35% to 80%) in the cement [4-8]. Research shows that, under critical temperatures (110 to 120°C), the hydrated calcium silicate gel (C-S-H) is prone to lattice transformation, forming dicalcium silicate hydrate (α -C₂SH) with large porosity and easy strength retrogression. By reducing the calcium/silica ratio, the hydrates can become xonotlite (C₆S₆H) and tobermorite (C₅S₆H₅) [10-12] with stronger thermal stability under high temperature conditions, which can avoid strength retrogression of set cement. When the temperature is over 160°C, however, the increase of SiO₂ content alone cannot eliminate the rapid dehydration and transformation of α -C₂SH lattice; and with the increase of temperature, the strength of set cement declines more obviously.

In summary, and given the strength retrogression of set cement at high temperatures in DHR, the geo-polymer raw materials have been chosen to adjust the phase ratio of CaO-SiO₂-Al₂O₃ in cement, which not only inhibits the strength retrogression of set cement at high temperatures but promotes the long-term development of strength. In addition, a new durable heat-resistant cement system has been developed which has supported the development of five wells like DHR GH-01 at Gonghe Basin of Qinghai province and provides a guarantee of cement slurry system for long-term and safe development of DHR at a later stage.

2. EVALUATION ON INHIBITION OF STRENGTH RETROGRESSION OF SET CEMENT

2.1 Experimental materials and methods

Through silica aluminum bonding enhancement, the geo-polymer materials are added in an alkaline, high temperature hydro-thermal environment to effect a hydro-thermal reaction to transform the hydrates from high-temperature cement and reduce the content of calcium hydroxide, so as to inhibit the strength retrogression of Portland cement at high temperatures.

(1) Experimental Materials

Grade G cement of Jiahua Company; HT (high temperature) strength stabilizer SCM; HT strength stabilizer SCKL; large temperature difference retarder SCR-4; fluid loss reducer SCF-180S; dispersant DZS; suspension agent DZW.

Two materials are chosen in this experiment by adjusting the phase ratio of CaO-SiO₂-Al₂O₃ in cement: HT stabilizers SCM and SCKL. Both are powder, 325 mesh. See Table 1 for their chemical compositions.

Table 1: Compositions of HT stabilizer materials for set cement, %.

Materials	SiO ₂	Al ₂ O ₃	CaO
SCM	66.72	14.2	9.98
SCKL	57.74	35.2	1.91

(2) Experimental method

The ultrasonic intensity analyzer is used to test the long-term strength development of the cement slurry added with stabilizer materials at 210°C.

Put the set cement in the HTHP (high-temperature and high-pressure) curing kettle; set the curing temperature at 300°C and the curing pressure at 21 MPa; cure the set cement for 48 h, 7 d, and 30 d; then core the sample, test the uniaxial compressive strength and Poisson's ratio of the set cement, and test the tensile strength of the set cement using the splitting test method.

Then observe the set cement cured at 150 to 300°C to analyze the micro-appearance and hydrate products using the scanning electron microscope and X-ray powder diffractometer.

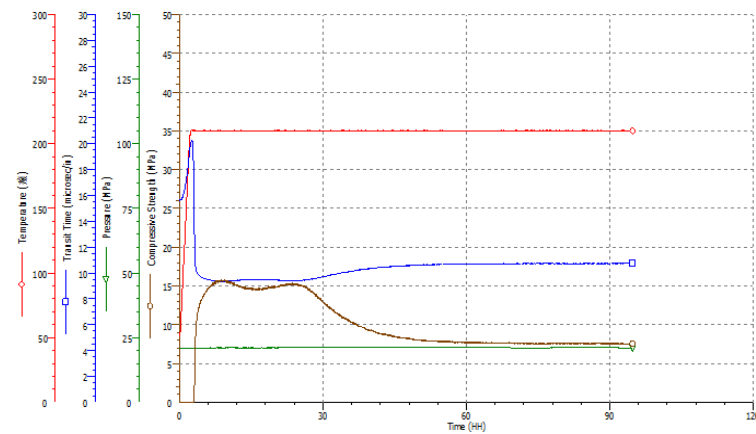
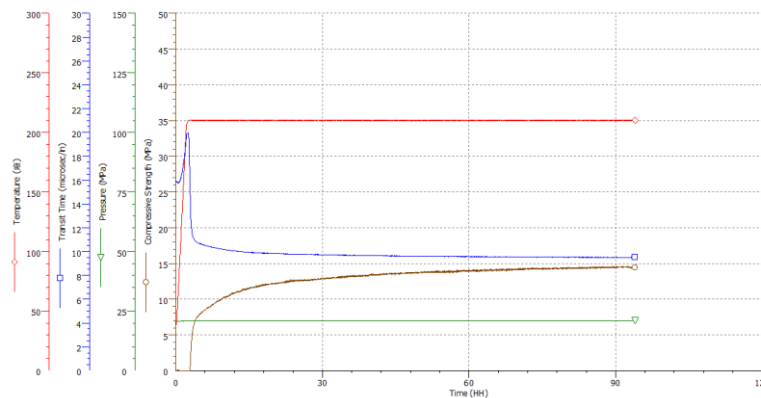
2.2 Evaluation of inhibition of set cement strength retrogression at high temperature

(1) Comparison of inhibition effect of different HT stabilizers

Add 50% (BWOC) of SCM and SCKL to the cement respectively; prepare the cement slurry at a water/cement ratio of 0.44; compare and observe the effect of different HT stabilizers on the long-term strength of set cement at 210°C. See Figure 1 and Figure 2 for the development of strength curves.

The SCM set cement contains a small amount of Al₂O₃; over 24 h, the set cement strength recovers a little bit, but the hydrates are not stable; after 48 h, the strength of set cement still declines, the set cement becomes looser and the grains inside the set cement are still divided. In contrast, the SCKL set cement has more Al₂O₃, which can react with calcium hydroxide in the set cement 24 h later in HT thermal-alkaline environment, producing calcium aluminum hydroxide silicate and improving the tightness of set cement, which not only inhibits the long-term decline of set cement strength but improves the long-term development of set cement strength, showing a trend of strength increase.

As a result, the SCKL is chosen to be further evaluated as HT stabilizer for set cement in the experiment.

**Figure 1: Development curve of set cement strength after adding SCM at 210°C.****Figure 2: Development curve of set cement strength after adding SCKL at 210°C**

(2) Effect of SCKL on compressive strength of set cement at 300°C

The effect of SCKL on the compressive strength of set cement at 300°C was observed. The formula of the cement slurry system is shown as follows: 400 g of Grade G cement + 320 g of HT strength stabilizer SCKL + 320 g of water; see Table 2 for the result. The set cement strength was up to 18.2 MPa at 300°C×48 h, and still increased after 7 d.

Table 2: Development of set cement strength at 300°C.

No.	Curing Conditions	Set cement strength MPa
1	300°C×48 h×21 MPa	18.2
2	300°C×7 d×21 MPa	19.7

(3) Effect of SCKL at different dosages on the long-term strength of set cement at 300°C

Add 40%, 60%, 80%, and 100% of HT stabilizer SCKL to the cement respectively, and cure for 30 d at 300°C; then observe the effect of different dosages of SCKL on the long-term HT mechanical properties of set cement. See Table 3 for the results. With the increase of dosages of HT stabilizer SCKL, the compressive strength of set cement after HT curing has been enhanced and the elastic modulus has increased; the compressive strength reaches the maximum at a dosage of 80% and the elastic modulus is less than 6 GPa.

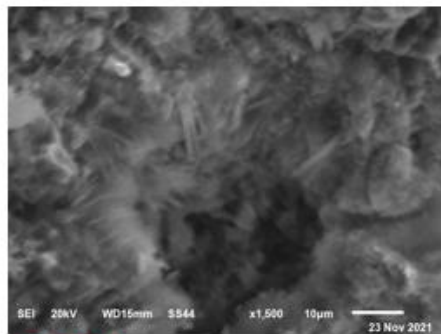
Table 3: Effect of different dosages of SCKL on mechanical properties of set cement at 300°C.

SCKL dosage %	Diameter mm	Length mm	Elastic modulus GPa	Compressive strength MPa	Poisson's Ratio
40	25.43	48.82	4.1912	16.018	0.146
60	25.33	49.06	4.2021	17.779	0.151
80	25.42	48.9	5.7999	20.216	0.178
100	25.37	48.86	7.4969	23.200	0.249

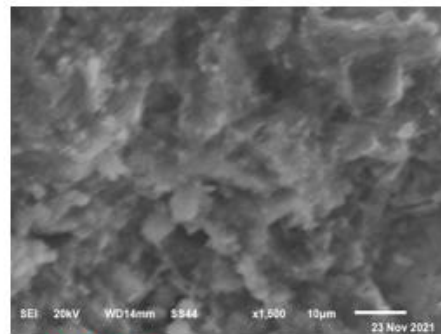
In summary, it is recommended that the optimum dosage of SCKL should be 80% (BWOC) which can serve the purpose of inhibiting long-term HT strength retrogress of set cement while maintaining a low elastic modulus in order to meet the requirement of airtightness for cold-water fracturing of DHR.

2.3 Effect of SCKL on micro-structure and hydrates of set cement

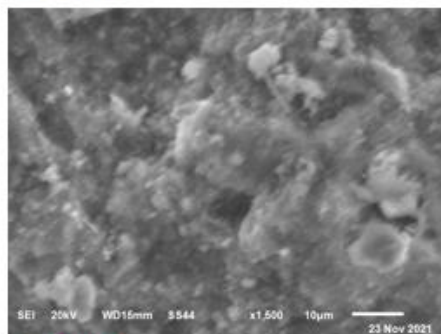
Analysis has been conducted on the micro-appearance of set cement at 150 to 300°C. See Figure 3 for the results. At 150°C, the hydrate products inside the set cement can be seen with obvious needle-shaped structures; at 180°C, the needle-shaped structures inside the set cement have decreased in quantity and have generated sheet structures; at 210°C, the set cement has no sheet structures inside and is relatively compact; at 300°C, rod-like structures occur inside the set cement and the hydrate products are compact; no peasting phenomenon is observed.



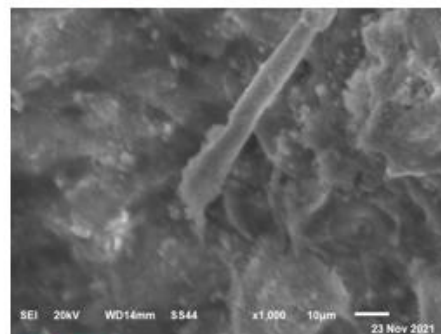
Micro-appearance inside the set cement at 150°C



Micro-appearance inside the set cement at 180°C



Micro-appearance inside the set cement at 210°C



Micro-appearance inside the set cement at 300°C

Figure 3: SEM analysis results of set cement at different temperatures.

XRD analysis was conducted on the hydrates of set cement after being cured at 150°C, 180°C, 210°C, and 300°C respectively. See Figure 4 for the hydrate products in set cement after HT curing. With the increase of temperatures, the tobermorite decreases in quantity in set cement; hydrates are transformed to hydrogrossular; at 300°C, the tobermorite disappears and becomes melilite, which inhibits the strength retrogression of set cement to some extent.

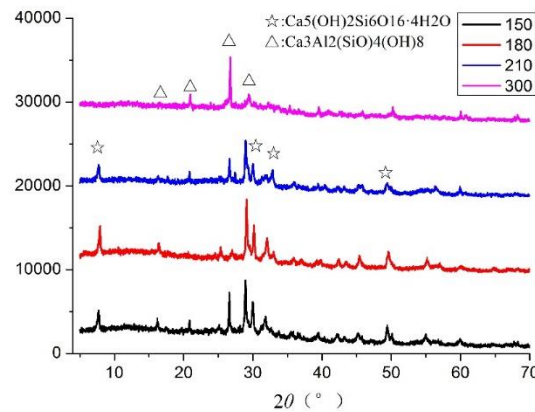


Figure 4: Effect of different temperatures on hydrates of set cement.

3. EVALUATION OF THE PROPERTIES OF THE DURABLE HEAT-RESISTANT CEMENT SYSTEM FOR DHR

It is required that there should be enough time for cementing jobs under high temperatures for DHR wells, so the HT retarder to be used and the subsidence stability should meet higher requirements.

3.1 Evaluation of HT retarder

SCR-4 is a kind of polymer retarder, which can effectively extend the thickening time of cement slurry under high temperatures without affecting the top strength of the cement sheath. We observed the effect of SCR-4 at different dosages on the thickening of the cement slurry system. See Table 4 for the results. With the increase of dosages of HT retarder SCR-4, the thickening time of cement slurry is extended. At a dosage of 10% to 11%, the thickening time of cement slurry is more than 4 h, which can guarantee a safe cementing job for DHR wells.

Table 4: Effect of retarder SCR-4 at different dosages on thickening time.

SCR-4 %	Thickening time (180°C×70 MPa) min		
	30 Bc	100 Bc	Transition time
8	156	159	3
9	166	168	2
10	263	264	1
11	377	378	1

3.2 Properties of HT heat-resistant cement slurry system

In view of high circulating temperatures (160 to 180°C) during DHR well cementing, and by adding fluid loss reducer, suspension agent, and retarder, the formula of HT cement slurry system is finalized as: Jiahua Grade G cement + 80% of HT strength stabilizer SCKL+ 4% of micro-silica + 6% of fluid loss reducer SCF-180s + 1.5% of dispersant DZS + 0.5% suspension agent DZW + 10% of retarder SCR-4 + water.

The basic properties of HT cement slurry system have been evaluated. See Table 5 for the results. The SCKL cement slurry has a density of 1.83 g/cm³ and has favorable rheology; at 180°C, its fluid loss is less than 50 mL and its settlement density difference is 0.03 g/cm³.

Table 5: Properties of SCKL cement slurry system.

Density g/cm ³	6-speed rheological readings at 93°C	Fluid loss at 180°C mL	Fluidity cm	Thickening (transition) time min	Settlement density g/cm ³
1.83	300+/220/151/77/4/3	46	20	378 (1)	1.81/1.84

See Figure 5 and Figure 6 for the thickening curve of HT SCKL cement slurry at 180°C and the shutdown-temperature variation thickening curve. At 180°C, the curve is at a right angle. The shutdown test finds that the cement slurry has very good settlement stability and can meet the requirement of safe cementing of liners in DHR wells at a temperature difference of 100°C.

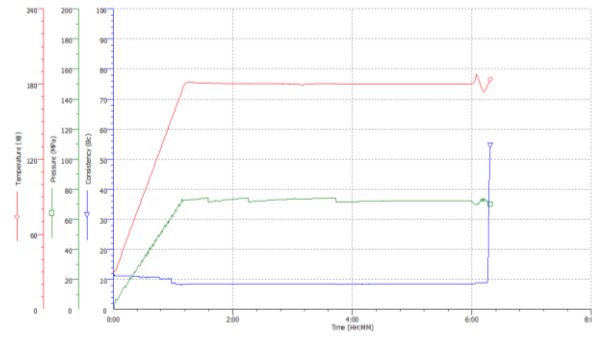


Figure 5: Thickening curve of SCKL cement slurry at 180 °C.

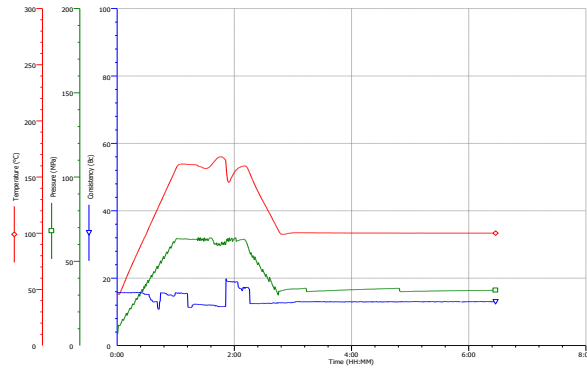


Figure 6: Shutdown-temperature variation test at HT shutdown for SCKL cement slurry system.

4. FIELD APPLICATION

The Gonghe Basin in Qinghai province was developed by well groups of one injector and two producers. The static temperatures were between 176 °C and 209 °C. Cooled by pilot mud, the circulating temperatures during cementing were between 160 °C and 180 °C. The cement system was used in five wells like Well GH-01. See Table 6 for more detail.

Table 6: Basic information on wells using the slurry system at Gonghe Basin in Qinghai province.

Geothermal area	Well No.	Well depth m	Bottomhole temperature °C	Circulating temperature °C	Cement density g/cm ³	Cementation quality
Gonghe DHR Basin in Qinghai	GH-01	4002.88	209	180	1.83	Good
	GH-02	4000.00	182	160	1.83	Good
	GH-03	4000.00	202	160	1.82–1.83	Good
	GH-04	4016.00	176	160	1.82–1.83	Good
	GH-05	4032.00	190	160	1.82–1.83	Good

The first injector-producer pilot well for DHR in China, Well GH-01, was successfully completed in October, 2019. The completion depth was 4002.88 m, and the bottomhole temperature was 209 °C. See Figure 7 for the casing program. The drilling fluid density prior to cementing was 1.17 g/cm³. The one-time cemented section of this well was 3620 m. The well was completed by cementing to the top of the screen pipe and had a very complex string structure.

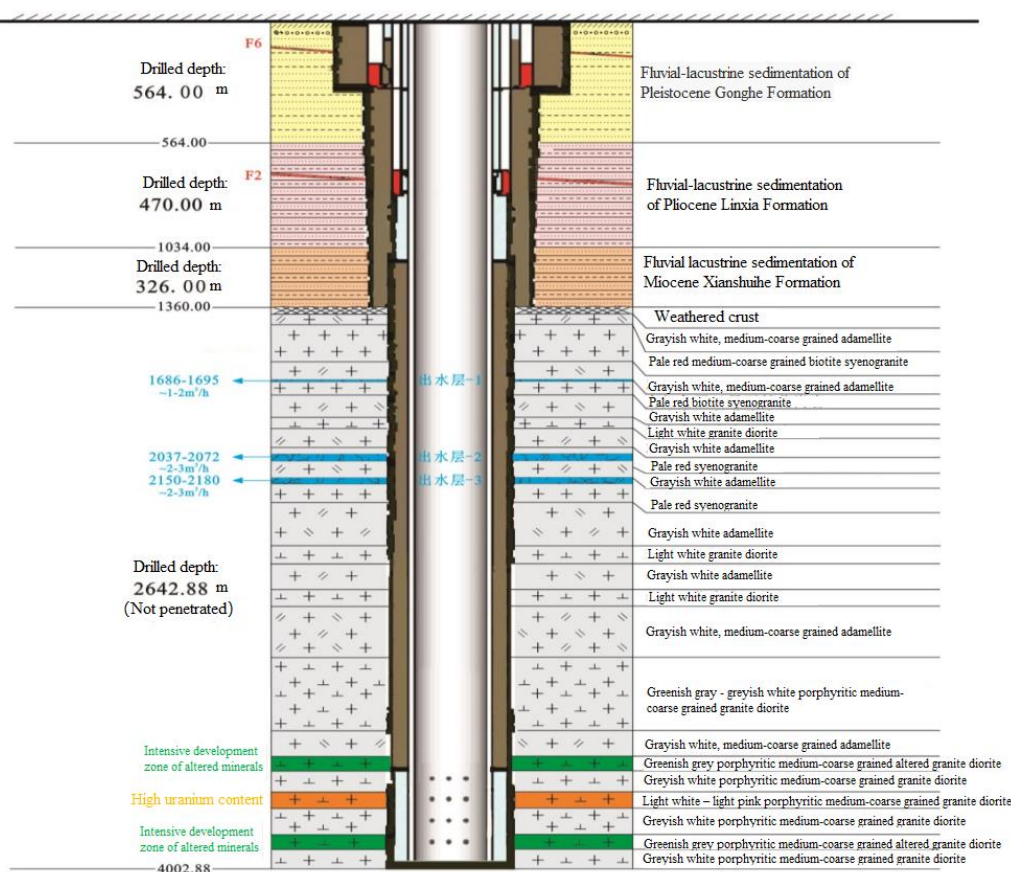


Figure 7: Casing program for Well GH-01.

The durable heat-resistant cement slurry system was used for this well, which was designed as a double-setting cement slurry system. Injected 42 m^3 of 1.83 g/cm^3 lead slurry and 24 m^3 of 1.83 g/cm^3 tail slurry. Injected 40 m^3 of fresh water with a density of 1.02 g/cm^3 at a rate of $1.0 \text{ m}^3/\text{min}$; injected 24 m^3 of heavy fluid with a density of 1.40 g/cm^3 ; displaced 1.90 m^3 of the fluid at a rate of 0.3 to $0.5 \text{ m}^3/\text{min}$ till pump pressure increased rapidly. The cementing process was successful; drilled the cement plug after 72 h to the reserved 10 m section, then conducted cement bond log (CBL). See Figure 8. The cementation job in this HT hole section was high quality.

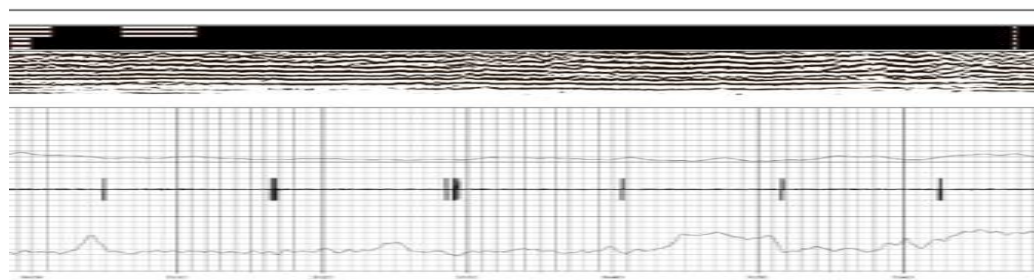


Figure 8: CBL quality for HT section in Well GH-01.

5. CONCLUSIONS

(1) By adjusting the phase ratio of $\text{CaO-SiO}_2\text{-Al}_2\text{O}_3$ in cement, the HT strength stabilizer, SCKL, has been formed for set cement, which not only inhibits the strength retrogression of set cement at 210°C but promotes the long-term development of set cement strength.

(2) We observed the effect of SCKL on HT mechanical properties of set cement at a condition of $300^\circ\text{C} \times 30 \text{ d}$; with an addition of 80% of SCKL, the compressive strength of set cement was 18.2 MPa for 48 h under 300°C ; when 100% of SCKL is added, the strength was 23.2 MPa for 30 d .

(3) In view of DHR wells at Gonghe Basin in Qinghai province, and by adding HT retarder, a new durable heat-resistant cement slurry system has been developed, which has a fluid loss of less than 50 mL at 180°C and a settlement density difference of 0.03 g/cm^3 ; its rheological and thickening properties are very good.

(4) This slurry system has been tested in five DHR wells at Gonghe Basin, Qinghai province, including the Well GH-01, and the cementation quality is good, which provides a guarantee of cement slurry system for long-term and safe development of DHR at a later stage.

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